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HARRY DIAMOND LABS ADELPHI MD
AN EVALUATION OF CURRENTLY USED PRINTED CIRCUIT BOARD MATERIALS--ETC(U)
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May 1981

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**An Evaluation of Currently Used Printed Circuit Board
Materials and Processes**

by Adolphe J. Edwards



**U.S. Army Electronics Research
and Development Command
Harry Diamond Laboratories**

Adelphi, MD 20783

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20. ABSTRACT (Continue on reverse side if necessary and identify by block number) A project was undertaken to evaluate currently used printed circuit board materials and processes. The procedure was to use combinations of the different types of commercially available photoresists and etchants to determine their fine line production capabilities. In the evaluation, the line widths were measured and compared with those of the artwork; also, the extent of undercutting was measured. From these data, a minimum line width limit was determined for each photoresist/photoresist thickness/etchant combination.		

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1. INTRODUCTION

Current printed circuit board fabrication practice tacitly assumes a lower limit for etched lines and spaces of roughly 10 mils (0.254 mm). One important consequence of this limitation is that it allows only one conductor line to be placed between adjacent pins of a standard microcircuit package. The resultant increase in circuit size due to the need for alternative routing paths is enormous. Even a modest improvement, such as two conductor lines between adjacent pins, would result in considerable space savings. Current practice includes a number of different materials and processes which, of course, have different limitations. A program was established to evaluate these materials and processes to determine their fine line resolution capabilities. There are numerous commercially available photoresists, both liquid and dry film, that are currently used in printed circuit board processing. Likewise, there are numerous etchants, many of which are proprietary, that are used in this processing. A determination had to be made of the significant differences between the various photoresists and the various etchants so that the program would remain of manageable size. Thus, the approach was to evaluate representative photoresist/photoresist thickness/etchant combinations and to determine their ultimate fine line production capabilities.

2. WORK PLAN

The plan was to include the various types of commercially available photoresist, which can be categorized as follows:

- a. Negative, dry film (NDF)
- b. Positive, liquid, high-solid content (PLH)
- c. Positive, liquid, low-solid content (PLL)
- d. Negative, liquid, high-solid content (NLH)
- e. Negative, liquid, low-solid content (NLL)

Each of these photoresists was to be evaluated at two different thickness values to determine

any effects of thickness. Early in the planning stages, however, it became apparent that the NLH photoresist could not be procured in time for inclusion in the program.

Included in the plan were five commercially supplied etchants, which can be categorized as follows:

- a. Ammonia base 1
- b. Ammonia base 2
- c. Ferric chloride base 1
- d. Ferric chloride base 2
- e. Chromic-sulfuric acid base

The purpose in including two etchants of the same type was that different additives are often used to provide different etching characteristics.

In summary, four different photoresists, each applied in two different thickness values, were to be used with each of five different etchants to yield 40 etched boards for evaluation. Two methods of evaluation were to be used:

- a. Measurements of the line and gap widths and comparisons of these with those of the artwork.
- b. Microscopic examinations of cross sections of etched lines and determinations of undercutting and etch factors.

3. PROCEDURE

The test specimen blanks 12 x 12 in. (30 x 30 cm) single-sided, one once copper-clad epoxy fiberglass boards. Holes for supporting the boards were drilled into those boards to be dip coated with liquid photoresists. Prior to coating, the copper surfaces were cleaned by scrubbing with a rotating fiber brush and an abrasive cleaner.

The dry film photoresists were applied with a hot press laminator. The liquid photoresists were applied by using a bench top dip coater.

The blanks, supported by a hanger, were dipped into a tank filled with liquid photoresist and withdrawn at a constant rate. The rate of withdrawal determined the coating thickness. The thicknesses of both the dry film and the liquid photoresist coatings were subsequently measured by the electron backscatter method.

The test patterns were 10×10 in. (25.4 \times 25.4 cm) square and contained lines with nominal widths of 10, 5, 2.5, and 1.25 mils (0.254, 0.127, 0.063, and 0.032 mm). Assorted circles, coils, bow ties, etc., were included in the test patterns to aid in the evaluations. Pattern exposures were made on a commercial exposure frame using a 400-W mercury vapor lamp. Initially, step plates were made for each photoresist thickness to determine proper exposure times. Minimum exposure times were used for the actual test specimens to minimize the effects of light scattering and heating. Pattern developing was performed in accordance with the individual manufacturer's recommendations.

Etching was carried out in a commercial vertical spray etching machine. For each etching run, the machine sump was filled with the etching solution, and 4 hr was allowed for the temperature to stabilize to that recommended by the manufacturer. The timer was then set at the value calculated from the metal thickness and the manufacturer's estimated etching rate. When necessary, 5-s increments were added until etching was deemed complete by visual observation. The etching operations were terminated when spaces between the test lines had cleared even though larger blank areas might not have cleared. The test specimens were then removed, rinsed thoroughly with running water, dried, stripped of photoresist, rinsed thoroughly, and dried a final time. Between etching runs, the spray etching machine was drained, rinsed thoroughly, neutralized, and again rinsed thoroughly.

The individual line and gap widths were then measured. First, an overall cursory examination

of the test specimens was made. The coordinates of the line edges were measured on a coordinate measuring table. The line and gap widths were then computed by successive subtractions of these coordinates. The line and gap widths of the artwork used to produce the test patterns had been similarly determined for comparison.

For microscopic examination and measurement of undercutting, sections were cut from the test specimens to include some of the line cross sections. For comparison, some sections contained lines oriented radially to the center of the test pattern, and some sections contained lines oriented tangentially to the center. The sections were mounted in epoxy and prepared by polishing through a series of successively finer grit polishing papers. Final polishing was performed on felt cloths impregnated with aluminum oxide slurries. The specimens were then viewed at 65X, 200X, and 1000X magnifications under a microscope, and the thickness and the undercutting of the lines were measured. Photomicrographs were made of representative line cross sections to illustrate the various phenomena observed.

4. EVALUATION

The differences in the line and gap widths of the artwork and the test specimens were tabulated. Then the average of these data were tabulated (table 1) and examined for any trends that might exist. Tabulations of the same data for lines oriented radially and those oriented tangentially to the centers of the pattern were made (table 2) and also examined for trends. Finally, tabulations of these same data for the upper and lower halves of the specimens were made (table 3) and examined. These average line width reductions (ΔW) are considered to be inherent for each photoresist/photoresist thickness/etchant combination.

From the thickness and undercutting measurements made on the line cross sections, etch factors (metal thickness/undercut) were

TABLE 1. AVERAGE DIFFERENCE BETWEEN LINE WIDTHS OF ETCHED PRINTED CIRCUIT BOARDS AND ARTWORK OVER ENTIRE BOARDS

Photoresist	Nominal thickness value (μm)	Ammonia base		Ferric chloride base		Chromic-sulfuric acid base	
		Etchant 1	Etchant 2	Etchant 1	Etchant 2	Etchant 1	Etchant 2
Negative, dry film	2000	-2.0	-1.9	-1.4	-1.5	-1.8	-2.0
	1000	-1.8	-1.7	-2.0	-2.5	-2.4	
Positive, liquid, high-solid content	260	-0.7	-1.4	-1.7	-1.4	-1.4	
	170	-1.0	-1.3	-1.2	-1.6	-1.1	
Positive, liquid, low-solid content	150	-1.0	-1.8	-1.5	-1.7	-1.6	
	65	-0.4	-0.8	-1.9	-1.5	-1.8	
Negative, liquid, low-solid content	25	+0.4	-0.1	-0.8	-0.4	-0.8	
	10	-0.2	-1.0	-1.7	-0.7	-1.3	

Note: Values are in mils

TABLE 2. AVERAGE DIFFERENCE BETWEEN LINE WIDTHS OF ETCHED PRINTED CIRCUIT BOARDS AND ARTWORK FOR RADIALLY AND TANGENTIALLY ORIENTED LINES

Photoresist	Nominal thickness value (μm)	Ammonia base				Ferric chloride base				Chromic-sulfuric acid base			
		Etchant 1	Etchant 2	Etchant 1	Etchant 2	Etchant 1	Etchant 2	Etchant 1	Etchant 2	Etchant 1	Etchant 2	Etchant 1	Etchant 2
Negative, dry film	2000	-2.0	-1.8	-1.5	-1.8	-1.5	-1.0	-1.8	-1.8	-1.0	-1.8	-1.1	
	1000	-1.6	-2.0	-1.5	-1.9	-2.0	-2.0	-2.4	-2.4	-2.3	-2.3	-2.4	
Positive, liquid, high-solid content	260	-1.1	-1.2	-1.7	-2.1	-0.6	-2.0	-1.8	-1.7	-1.1	-1.9	-1.6	
	170	-0.4	-0.3	-0.9	-0.7	-1.6	-2.2	-1.4	-1.4	-1.9	-1.6		
Positive, liquid, low-solid content	150	-0.8	-0.6	-1.6	-1.3	-1.6	-1.7	-1.2	-1.5	-1.4	-1.4		
	65	-1.3	-1.1	-1.4	-1.3	-1.1	-1.3	-1.5	-1.6	-1.0	-1.2		
Negative, liquid, low-solid content	25	+0.3	+0.7	-0.1	0.0	-0.6	-0.7	-0.3	-0.4	-0.9	-0.9	-0.8	
	10	-0.1	-0.2	-1.3	-1.4	-1.9	-1.5	-0.7	-0.7	-0.6	-0.6	-1.0	

Note: Values are in mils

TABLE 3. AVERAGE DIFFERENCE BETWEEN LINE WIDTHS OF ETCHED PRINTED CIRCUIT BOARDS AND ARTWORK
FOR UPPER AND LOWER HALVES OF BOARD

Photoresist	Nominal thickness value (μin.)	Ammonia base				Ferric chloride base				Chromic-sulfuric acid base			
		Etchant 1		Etchant 2		Etchant 1		Etchant 2		Etchant 1		Etchant 2	
		Upper	Lower	Upper	Lower	Upper	Lower	Upper	Lower	Upper	Lower	Upper	Lower
Negative, dry film	2000	-2.0	-1.9	-1.8	-1.8	-1.2	-1.6	-1.1	-1.7	-2.2	-1.4	-2.4	-2.3
	1000	-1.9	-1.9	-1.6	-1.8	-1.9	-2.1	-2.3	-2.7	-2.4	-1.2		
Positive, liquid, high-solid content	260	-1.7	-0.6	-2.2	-1.4	-2.5	-1.0	-2.6	-1.0	-2.4	-1.2		
	170	-0.5	-0.3	-1.0	-0.5	-1.8	-1.9	-1.8	-1.4	-2.2	-1.3		
Positive, liquid, low-solid content	150	-1.2	-0.2	-1.7	-1.1	-1.8	-1.5	-2.1	-2.7	-2.4	-2.3		
	65	-1.6	-0.8	-1.6	-1.1	-1.5	-1.0	-1.9	-0.6	-1.6	-1.1		
Negative, liquid, low-solid content	25	+0.2	+0.5	0.0	0.0	-0.8	-0.6	-0.7	-0.2	-1.0	-0.6		
	10	-0.4	0.0	-1.1	-0.7	-1.6	-1.9	-0.9	-0.6	-1.3	-1.4		

Note: Values are in mils

calculated (table 4). Considering that even if the etching process could be stopped at the precise instant of vertical penetration so that no reduction in the width of the bases of the lines occurred, there remains a limitation on the minimum line width due to the undercut. If the assumption is made that this minimum occurs when the undercut equals one half the line width, then this limitation can be related to the metal thickness and etch factor by

$$\begin{aligned} \text{limit} &= 2 \times \text{undercut} \\ &= 2 \times \text{metal thickness/etch factor.} \end{aligned}$$

Combining ΔW with the values given by the above equation gives calculated values of the ultimate line width limits

$\Delta W + (2 \times \text{metal thickness})/\text{etch factor}$
for the various photoresist/photoresist thickness/etchant combinations shown in table 5.

TABLE 4. ETCH FACTORS CALCULATED FROM MEASURED COPPER THICKNESS AND UNDERCUT

Photoresist	Nominal thickness values (μin.)	Ammonia base		Ferric chloride base		Chromic-sulfuric acid base
		Etchant 1	Etchant 2	Etchant 1	Etchant 2	
Negative, dry film	2000	1.4	1.5	1.6	1.6	1.4
	1000	1.3	1.8	2.4	1.6	2.2
Positive, liquid, high-solid content	260	2.4	4.8	6.5	4.8	2.3
	170	3.3	5.6	9.7	6.6	3.3
Positive, liquid, low-solid content	150	4.1	3.5	14.0	7.0	2.4
	65	4.5	4.8	14.5	5.4	4.4
Negative, liquid, low-solid content	25	2.3	2.9	13.5	4.3	5.0
	10	2.7	3.8	6.0	3.6	2.9

Note: Values are in mils.

TABLE 5. CALCULATED VALUES OF MINIMUM LINE WIDTHS FOR 1-OZ COPPER ETCHED PRINTED CIRCUIT BOARDS

Photoresist	Nominal thickness values (μin.)	Ammonia base		Ferric chloride base		Chromic-sulfuric acid base
		Etchant 1	Etchant 2	Etchant 1	Etchant 2	
Negative, dry film	2000	4.0*	3.8*	3.2*	3.3*	4.0*
	1000	4.0	3.3*	3.2*	4.3	3.7*
Positive, liquid, high-solid content	260	2.2	2.4	1.9	2.3	2.8
	170	1.2	1.3	2.2	1.9	2.6
Positive, liquid, low-solid content	150	1.4	2.2	1.9	1.8	2.6
	65	1.6	1.9	1.4	2.1	1.7
Negative, liquid, low-solid content	25	0.8	1.1	1.0	1.0	1.4
	10	1.2	1.7	2.2	1.5	2.3

*Limitations of these combinations are greater because of photoresist detachment during developing

Note: Values are in mils.

5. RESULTS

Thickness measurements on the dry film photoresists verified the listed value for the 1-mil (0.0254-mm) material, but were low for the 2-mil (0.0508-mm) material, probably because the thickness exceeded the range of the isotope used (table 6). Measurements indicated that both materials were uniform in thickness. The dip coating process used to apply the liquid photoresists yielded thickness profiles with maxima just below the vertical centers of the specimens. The thickness values obtained were inversely proportional to the withdrawal rates, with the faster withdrawal rates giving thicker but more uneven coatings. The average thickness values for the upper and lower halves of the specimens are listed for each liquid photoresist in table 6.

The step plates for each photoresist thickness were examined, and exposure times were selected to give adequate exposure but to avoid overexposure as follows:

NDF		
2 mils	1 s	
1 mil	1 s	
PLH		
225 μ in.	56 s	
145 μ in.	20 s	
PLL		
160 μ in.	37 s	
65 μ in.	24 s	
NLL		
20 μ in	18 s	
8 μ in	6 s	

The exposed photoresist patterns were developed according to the manufacturer's recommendations. While developing the dry film photoresists, some of the finer lines became detached. For the 2-mil dry film photoresist, lines less than 7 mils (0.1778 mm) in width were lost during the developing process. For 1-mil dry

TABLE 6 PHOTORESIST THICKNESS MEASUREMENTS

Photoresist	Nominal thickness value (μ in.)	Photoresist thickness (μ in.)		
		Upper half of board	Lower half of board	Av
Negative, dry film	2000	1680*	1680*	1680*
	1000	860	860	860
Positive, liquid, high solid content	260	140	310	225
	170	135	155	145
Positive, liquid, low solid content	150	145	195	160
	65	60	70	65
Negative, liquid, low solid content	25	15	25	20
	10	5	10	8

*Thickness beyond range of isotope used

Note Values are in mils

film photoresist, lines less than 4 mils (0.1016 mm) in width were lost during the developing process. Thus, there are apparently limitations on the fine line resolution (listed in table 5) due to the photoresist thickness/developing process.

For the etching operations, it was intended that the volume of the etchants be large enough that the concentrations would remain essentially constant so that the etching rates would remain constant throughout each etching series. However, the etching time increased as more specimens were etched in any given etchant. The range of etching times for each etchant is as follows:

Ammonia base	
Etchant 1	65 to 70 s
Etchant 2	125 to 130 s
Ferric chloride	
Etchant 1	65 to 80 s
Etchant 2	75 to 110 s
Chromic-sulfuric acid	80 to 90 s

Portions of the test patterns near the tops of the liquid photoresist coated specimens were lost during etching. Apparently the photoresist in these areas was not thick enough to withstand the etchant. A thickness greater than 50 μ in. (1.270 μ m) for the positive, liquid photoresist and 5 μ in. (0.1270 μ m) for the negative, liquid photoresist appears necessary to withstand the etchants.

Evaluation of the data in tables 1 to 3 showed that in practically all instances the line widths of the test specimens were less than those of the artwork from which they were produced. There was less line width reduction for the two ammoniacal etchants than for the three acidic etchants. A ranking of the etchants in order of increasing line width reduction would be ammonia base 1, ammonia base 2, ferric chloride base 1, ferric chloride base 2, and chromic-sulfuric acid base.

Also, effects of photoresist thickness on line width reduction can be seen in tables 1 to 3. There was no appreciable difference in line width reduction for the 1- and 2-mil NDF photoresist specimens, except for those etched with the two ferric chloride etchants. For those specimens, the thicker photoresist gave less reduction. Of the liquid photoresist specimens, only the PLH and NLL specimens showed significant differences in line width reduction for the different thicknesses and these only for the two ammoniacal etchants. The 160- μ in. (4.06- μ m) PLH specimens had slightly less reduction than did the 230- μ in. (5.842- μ m) specimens, and the 25- μ in. (0.635- μ m) NLL specimens had slightly less reduction than the 10- μ in. (0.254- μ m) specimens. Considering the relative thicknesses of the different photoresist coatings, the thinner coatings were associated with less line width reduction. A ranking of the different photoresists in increasing order of line width reduction would be NLL, PLL, PLH, and NDF. This, incidentally, is the order of increasing thickness. The fact that the thinner PLH photoresist specimens had slightly less line width reduction than the thicker ones while the reverse was true for the NLL specimens suggests that there might be an optimum thickness for each type of photoresist coating. Plots of line width reduction for the different photoresists are shown in figure 1.

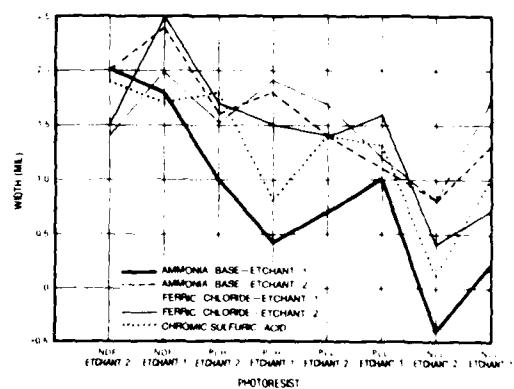


Figure 1. Line width reduction of etched lines.

There were no discernible differences in line width reductions for the lines oriented radially and those oriented tangentially to the center of the patterns. There was definitely less line width reduction for the lower halves of the liquid photoresist specimens than for the upper halves (table 3). These differences were noted for all etchants and are obviously related to photoresist thickness. A minimum thickness of each type of photoresist is necessary to withstand the etchants. At this minimum value, line width reduction is probably rather severe, but it decreases as the photoresist thickness increases. After the photoresist thickness increases beyond some optimum value, the line width reduction increases again due, most likely, to a complexity of light scattering processes. These processes include divergence, reflection, and diffraction, and the evaluation of them is beyond the scope of this investigation. Suffice it to say that the optimum thickness of each photoresist is some value between that thickness just barely sufficient to effectively withstand the etchants and that thickness where the effects of light scattering become measurably deleterious.

Microscopic examinations of the cross sections of lines formed by etching revealed some distinct characteristics. The edges of lines etched with the ammoniacal etchants were slanted considerably from the vertical (fig. 2, 3), thus indicating an appreciable lateral etching rate. Of these, the NDF specimen line edges (fig. 2) were almost straight, a fact that indicates a uniform lateral etching rate over the thickness of the metal. The edges of the lines formed with the liquid photoresists were somewhat concave (fig. 3). Their being concave indicates a slower horizontal etching rate near the surface of the

metal. This difference in the surface lateral etching rate is probably due to photoresist adhesion or stiffness or both. The extent of undercutting is slightly less for ammonia base etchant 1 than for ammonia base etchant 2, as can be seen from the etch factors in table 4.

The edges of lines of specimens etched with the acidic etchants (fig. 4 to 6) were more nearly vertical than those etched with the ammoniacal etchants. Of these, the NDF specimens (fig. 5) were somewhat slanted; the slanting indicates a relatively faster lateral etching rate. This difference, again, is probably due to differences in photoresist adhesion or stiffness or both. The line edges of all liquid photoresist specimens were nearly vertical, except the 10- μ in. NLL, which thickness is considered marginal. This fact indicates a relatively slower lateral etching rate. Among the acidic etchants, the extent of undercutting, which is a measure of lateral etching rate, was least for ferric chloride etchant 1 and greatest for the chromic-sulfuric acid etchant.

A possible explanation for the slower lateral etching rates or higher etch factors for the ferric chloride etchants is that they have a lower tolerance for dissolved copper. Thus, they are much slower acting at high copper concentrations. This could well be the situation at the surface of the copper, where the etchant circulation is restricted by the photoresist layer. In fact, many instances were noted where lateral etching had advanced farther below the surface than at the surface of the copper (fig. 7 to 9). This phenomenon is thought to be the origin of slivers, which are a problem in the fine line etching of printed circuit boards.

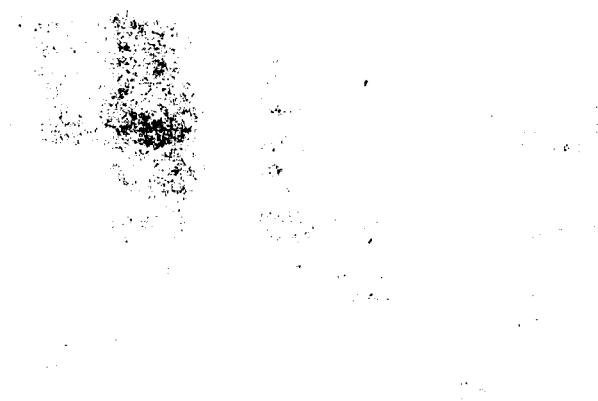


Figure 2. Cross section of NDF specimen line etched with ammonia base etchant 2; line edges are almost straight and slanted considerably from vertical. 200X.

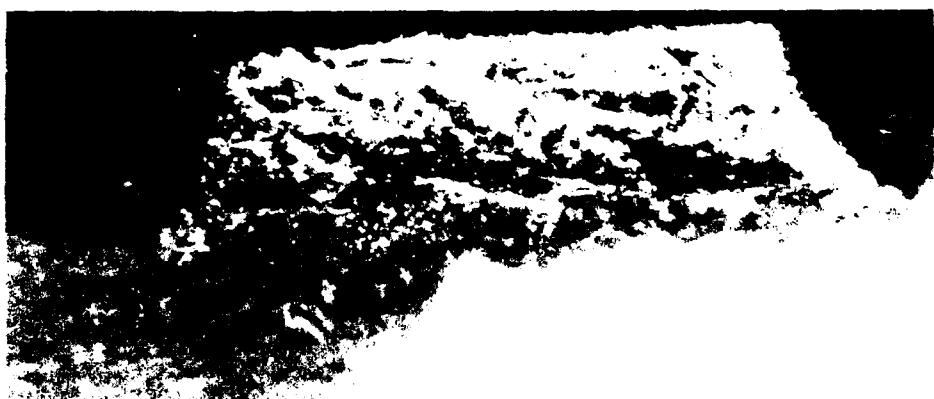


Figure 3. Cross section of PLL specimen line etched with ammonia base etchant 1; line edges are slanted considerably from vertical and are concave. 1000X.



Figure 4. Cross section of PLL specimen line etched with ferric chloride etchant 1; line edges are nearly vertical. 1000X.



Figure 5. Cross section of NDF specimen line etched with ferric chloride etchant 2; line edges are slanted from vertical slightly more than for ferric chloride etchant 1; 65X.



Figure 6. Cross section of NLL specimen line etched with chromic-sulfuric acid etchant; line edges are nearly vertical and are undercut somewhat more than for ferric chloride etchants, but less than for ammonia base etchants; 65X.

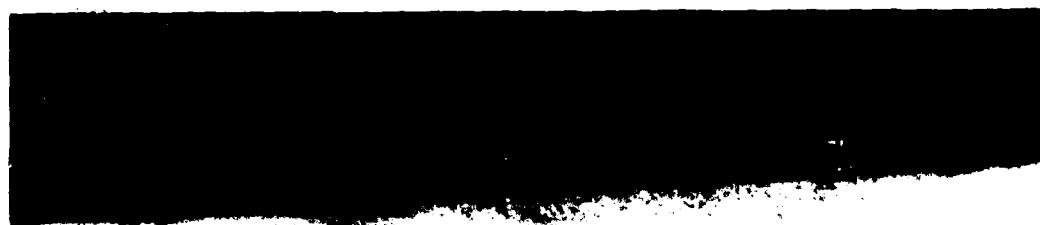


Figure 7. Cross section of fine lines etched ferric chloride etchant 2; advanced lateral etching below surface of copper; 200X.



Figure 8. Cross section of fine lines etched with chromic-sulfuric acid etchant; almost detached sections of metal (slivers) at tops of lines; 200X.



Figure 9. Cross section of fine line etched with ferric chloride etchant 1; metal (sliver) at top of line is almost detached; 1000X.

6. CONCLUSIONS AND RECOMMENDATIONS

The fine line resolution achievable in copper-clad printed circuit boards depends on a multiplicity of factors such as these:

- Metal thickness
- Photoresist type
- Photoresist adhesion
- Exposure conditions
- Artwork acuity
- Developing conditions
- Etching conditions

These factors interact usually to yield a reduction in line width (ΔW) below that of the artwork. These factors can be controlled to some degree as can be seen by the variations in ΔW for the different combinations. A further limitation on the line width is due to the degree of undercutting or lateral etching. When the lateral etching from each side approaches one half the line widths, any of several problems such as photoresist detachment or sliver formation may arise. The combination of the line width reduction (ΔW) and twice the extent of lateral etching equals the ultimate line width limit as tabulated in table 5. Because there are some uncertainties, the values are rounded off, usually to the next higher half mil.

Based upon the values in table 5, the following are offered as lower width limits for the

various photoresist/photoresist thickness/etchant combinations when used to process 1-oz (28-g), 1.4-mil (0.036-mm) copper-clad printed circuit boards. These values are intended to represent the design limits for line widths, and it is suggested that discontinuities could result for line widths designed below these limits. No consideration was given to current carrying capacity or the ohmic resistance of the lines, which might well further restrict these limits.

a. The line width limits are restricted by the developing process to 7 mils for the 2-mil NDF photoresist and to 4 mils for the 1-mil NDF photoresist, regardless of etchant.

b. The 170- μ in. PLH photoresist has a line width limit of 1.5 mils with the ammoniacal etchants and 2 mils with the acidic etchants. The 260- μ in. PLH photoresist has a line width limit of 3 mils with the chromic-sulfuric acid etchant, 2.5 mils with either ferric chloride etchant 2 or the two ammonical etchants, and 2 mils with ferric chloride etchant 1.

c. The line width limit is 1.5 mils for the 65- μ in. PLL photoresist with ferric chloride etchant 1 and 2 mils with other etchants. The line width limits for the 150- μ in. PLL photoresist are 1.5 mils with ammoniacal etchant 1, 2 mils with both ferric chloride etchants, and 2.5 mils with ammoniacal etchant 2 and the chromic-sulfuric acid etchant.

d. The 10- μ in. NLL photoresist has a line width limit of 2.5 mils with ammoniacal etchant 2 and ferric chloride etchant 1, 2 mils with ferric chloride etchant 2 and the chromic-sulfuric acid etchant, and 1.5 mils with ammoniacal etchant 1. The 25- μ in. NLL photoresist has a line width limit of approximately 1 mil with both ammoniacal and both ferric chloride etchants and 1.5 mils with the chromic-sulfuric acid etchant.

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